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**JP-A-50 030 708
JP-A-53 018 598
US-A-3 478 015**

**CHEMICAL ABSTRACTS, vol. 89, 1978, abstract no.
126336v, page 332 Columbus Ohio (US)
JOURNAL OF AGRICULTURAL FOOD CHEMISTRY, vol.
18, no. 5, 1970, Washington (US) P.E. KOEHLER et al.:
"Factors Affecting the Formation of Pyrazine
Compounds In Sugar-Amine Reactions", pages 895-898**

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Description

The present invention relates to cooked flavors, and more specifically to cooked flavors suitable for use in smoking compositions. The invention is also concerned with processes for preparing cooked flavors and incorporating them into smoking compositions, particularly those having a high content of tobacco stems, to produce a more acceptable and flavorful smoking product.

In general, tobacco stems and midribs represent an unsatisfactory portion of the cured tobacco leaf, from the standpoint of smoking, in the sense that they produce a stemmy taste on smoking and lack the desirable aroma and taste generally associated with tobacco lamina.

Stems are usually separated from the desirable leaf or lamina of tobacco. To throw away the stems and midribs is uneconomical and, for this reason, methods have been devised to modify and make them usable in smoking products. One of the methods which has been found to be relatively satisfactory, because of its simplicity and low cost, is to pass the stems between rollers in order to break up the bulky, rigid structure and to produce rolled stems, which are a flattened, loosened product which can be shredded and blended with shredded tobacco leaf.

It has been found, however, that the rolled stems still impart a certain amount of the harshness and off-taste which are imparted by tobacco stems to the smoke from cigarettes or other tobacco products into which they are introduced. Stems from burley tobacco are particularly undesirable in this respect. As a consequence, only a small portion of the stems from tobacco used in filler for cigarettes or cigars can be rolled and blended back into the filler.

We have now discovered a means by which rolled stems may be used to produce tobacco products having a more acceptable smoke. Stems from both bright and burley tobacco treated by our process may be used in greater proportion to produce a cigarette which yields comparatively bland smoke.

The reaction of sugars with amino acids to produce desirable flavorants for smoking materials has generated increased interest. For example, US-A 3 478 015 describes "browning reactions" in which an amino acid and a sugar having an active carbonyl are reacted in a lower alkyl polyhydric alcohol solvent in the absence of water at a temperature less than 90°C for about 5 to 15 hours. Generally the amino acid to solvent ratio is in the range of 1 to 3% by weight and the sugar solvent ratio is in the range of 3 to 15% by weight. The reaction is carried out in such a manner that no free amino acid remains in the final product. The resultant reaction mixture is applied to tobacco to about 1% by weight of the tobacco.

US-A 3 920 026 describes tobacco flavorants prepared by reacting valine with a carbonyl compound selected from sugars, dihydroxy acetone, or pyruvaldehyde. The molar ratio of amino acid to carbonyl compound can be from about

1:0.5-10, with a ratio of 1:4-5 being optimum. The reaction takes place in a solvent such as glycerol or propylene glycol and at a temperature between about 120 and 200°C, and preferably at about 150 to 160°C for 0.5 to 5 hours depending on the type of sugar used. Flavanoid catalysts, such as quercetin or rutin or a hydroxyacid such as ascorbic acid, may be used. The reaction mixture may be applied directly onto tobacco or may be fractionated to separate the volatile and non-volatile fractions, which are then used as tobacco flavorants.

US-A 3 722 516 discloses the addition of dihydroxyacetone alone or in combination with amino acids to enhance the natural flavor characteristics of tobacco, and especially the caramellike or burned sugar-like aroma thereof.

JP-A 9239/71 discloses certain tobacco "perfumes" such as 1-deoxy-1-L-proline-D-fructose, which is a sugar-amino acid condensation product. This compound, as well as others, are prepared by reacting an amino acid with a sugar in an aqueous or alcohol solvent, and usually in the presence of an acid catalyst such as malonic acid. The desired compounds may be separated and purified by the use of ion-exchange resins and are thereafter applied to tobacco.

JP-A 3398/73 discloses certain other specific amino acid-sugars such as 1-deoxy-1-L-alanine-D-fructose that are synthesized from their respective components by the Amadori rearrangement, in the presence of malic acid as catalyst, by heating at 130°C for 15 minutes. The specific compounds are isolated and may be combined with cocoa to produce tobacco flavorants.

Two articles by Koehler (Journal of Agricultural Food Chemistry, Volume 17, Number 2, pages 393-396, 1969 and *ibid.*, Volume 18, Number 5, pages 895-989, 1970) discuss pyrazine formation in sugar-amino acid model systems. The normal model system consists of asparagine and glucose reactants in a 1:1 molar ratio. Diethylene glycol solvent and a small amount of water are also used as solvents. The mixture is heated for 24 hours at 120°C. The use of a base catalyst, such as sodium hydroxide or ammonium hydroxide, results in an increase in the production of volatile alkylated pyrazines. In some instances the addition of an aldehyde, such as acetaldehyde, appeared to increase pyrazine formation. Koehler's work was related to browning reactions known to occur in foods such as roasted peanuts, coffee, cocoa, and potato chips; and his primary object was to determine the chemical factors involved in volatile pyrazine formation during such reactions. Although volatile pyrazines were isolated and identified, there is no suggestion by Koehler that his reaction mixture would be suitable for use as a tobacco flavorant.

Chemical Abstracts, vol. 89 (1978), page 332 at 126 336 v discloses a process for synthesizing tobacco flavors by heating a mixture of ammonium ion and/or amino acids, fatty amines, fatty dicarbonyl compounds and/or sugars and nicotine.

The present invention now provides a process

of producing a cooked flavorant in which a reducing sugar is heated with ammonia and amino acid characterized in that the reducing sugar is reacted with a source of ammonia in a weight ratio of reducing sugar to ammonia calculated as ammonium hydroxide (30%) from 5 to 15:1, in the presence of only a trace amount of an amino acid or monoamide thereof in a weight ratio of sugar to amino acid from 200 to 300:1, and in the absence of any further reactant. The reaction mixture is preferably heated to a temperature in the range of 90°C to 105°C. Once the desired temperature range is attained, the heating may be continued for 5 to 15 minutes or, alternatively, the reaction mixture is removed from the heat source and cooled to room temperature. The thus prepared mixture has been found to produce desirable reaction products highly suitable for use in smoking products, and particularly those having a high content of tobacco stems. Application of such cooked flavorant to the stems at a level up to about 5% by weight of the stem produces an acceptable smoking product.

Although the reaction flavor product has been found particularly suitable for use in enhancing the flavor and aroma of tobacco stems, such cooked or reaction flavors are equally suitable for use on other smoking materials selected from tobacco leaf, reconstituted tobacco, non-tobacco smoking substitutes and mixtures thereof. The reaction flavors may be incorporated therein at a level from about 0.5 to 10% by weight based on the dry weight of the smoking material.

The use of such reaction flavors, particularly in tobacco compositions having a high stem content, results in a tobacco product having improved smoking characteristics. The flavorant-stem product is highly suitable for use in cigarettes at a blend level up to 50% if desired.

Reducing sugars are reacted with a source of ammonia in the presence of a trace amount of certain amino acids to produce flavorants suitable for use in smoking products. Suitable reducing sugars include glucose, fructose, mannose, galactose, and mixtures thereof, with syrups containing approximately 42% or more of fructose being preferred. Disaccharides and polysaccharides may also be used as the source of reducing sugar as long as they are converted to monosaccharides by conventional hydrolysis prior to reacting with the other named reagents. Saccharide mixtures such as corn syrup, malt syrup and invert sugar may also be used as long as conversion to monosaccharides precedes the reaction with amino acids.

Representative amino acids include aspartic acid and glutamic acid and the monoamides thereof, specifically asparagine, and glutamine. The weight ratio of sugar to amino acid will generally be in the range of 200-300:1 with a ratio of about 235-245:1 being preferred. The preferred source of ammonia for purposes of this invention is concentrated ammonium hydroxide. The weight ratio of sugar to concentrated ammonium hydroxide (30%) will generally be about 5-15:1

with 6-8:1 being preferred. It will be appreciated that alternate sources of ammonia may be employed, such as gaseous or liquid ammonia or subjectively acceptable ammonium salts. Suitable ammonium salts include ammonium orthophosphate, ammonium dihydrogen orthophosphate, diammonium monohydrogen orthophosphate, ammonium citrate, ammonium acetate and the like.

The reaction is generally carried out in a flask, preferably one equipped with a reflux condenser to prevent loss of desirable volatiles. The flask and contents may be heated in a steam bath, an oil bath, or the like, and the time necessary to reach the optimum temperature of about 90 to about 105°C is determined either by the size of the reaction vessel or the temperature of the heating system or both. A stainless steel jacketed vessel having a steam inlet and outlet, condenser and cooling coils may be used for larger scale reactions. In most instances it is only necessary to bring the reactants to the desired temperature, and then the reaction is stopped by removing the heat source. In certain instances it may be desirable to continue heating for about 5 to 15 minutes once the desired temperature is attained. Best results are obtained when the reaction mixture is actively cooled to about 49°C or lower. Cooling may readily be effected by diluting the reaction mixture with water, or, alternatively, the reaction mixture may be cooled by means such as cooling coils within the reaction vessel, an ice bath and the like.

Although the cooled reaction flavor product may be used without further dilution, it may, in certain instances, be desirable to dilute the flavor product with an acceptable diluent. The degree to which the reaction flavor product is diluted with water or other acceptable diluent is a matter of choice and may, to some extent, depend upon its ultimate use. Generally a 1:1 to about a 5:1 dilution with water is adequate and results in an aqueous reaction flavorant product readily adaptable for use on any type of smoking composition.

The term "smoking composition" includes all forms of tobacco such as shredded filler, leaf, stem, stalk, homogenized leaf cured, reconstituted cigar binder, pipe tobaccos, or reconstituted tobacco in sheet or predetermined forms, which are cast or extruded from "tobacco slurries". Rolled and cut tobacco stems in either expanded or unexpanded form are particularly preferred. In addition, smoking compositions may encompass the various smoking substitutes formulated from non-tobacco materials. The smoking substitute may be utilized alone or blended in varying proportions with other forms of tobacco. Representative formulations for non-tobacco smoking material may be found in US-A 3 529 602; US-A 3 703 177; US-A 3 796 222; US-A 4 019 521; US-A 4 079 742; and references cited therein.

The reaction flavors produced in accordance with the present invention may be applied to the smoking composition by direct spraying methods

known in the art. They may also be applied by dispersing or diluting further in a suitable carrier, water for example, or may be directly mixed or otherwise combined with the smoking material. When an aqueous carrier is employed, the mixture is air-dried or otherwise treated to remove the carrier. Generally the reaction flavor product will be incorporated into the smoking composition or a component thereof at a level of 0.5 to 10% by weight of the composition. It will be recognized that larger amounts of the reaction flavor product may be incorporated into a single tobacco component which is then blended with other filler materials to produce a smoking composition having the previously described level of reaction flavor product.

The reaction flavors can, if desired, be incorporated in cigarette paper or in filters. However, it will generally be more desirable to incorporate the flavors in the smoking composition itself.

In a preferred embodiment, the cooled reaction flavor is diluted approximately 1:1 with water and sprayed or otherwise applied to rolled, crushed tobacco stems which are subsequently shredded and blended with conventional tobacco filler at a level up to about 50% based on the total weight of the filler. Humectants and additional casing materials conventionally employed in tobacco processing may be incorporated with the reaction flavorant or applied separately to the stem material. Stems having the reaction flavor applied thereto, on smoking, have a smoother response and less "stemmy" taste. In addition, stems containing the reaction flavor may be incorporated at a higher level in a blend than previously possible.

The following examples are illustrative.

Example I

To a glass jacketed reaction vessel equipped with reflux condenser and stirrer/agitator were added the following reactants: 1278 grams of a syrup containing 72% fructose by weight (900 Isomerase Syrup, Clinton Corn Sweeteners), 3.9 grams DL-aspartic acid and 150 ml of concentrated (30%) ammonium hydroxide. The contents of the reaction vessel were stirred and heated by passing steam through the jacket until the temperature of the reaction solution reached 95°C. The steam was terminated and the contents of the vessel were removed and allowed to cool to room temperature. The yield of reaction flavor product was 1397.5 grams or 98.6%.

The cooled reaction mixture was sprayed on shredded, flue-cured tobacco stems to a level of 2.5% by weight. In addition, glycerine and propylene glycol were sprayed to a level of 2% each by weight of the stems. The stems were fabricated into cigarettes having conventional cellulose acetate filters attached thereto. Control cigarettes were made in a similar manner using untreated stems. A small panel of experienced smokers smoked and compare the treated stem cigarettes with the control and found the treated cigarettes to be less hot and harsh. Although a bitter after-

taste was noted, the treated cigarettes seemed smoother and less peppery than the control.

Example II

In a manner similar to Example I, 314 grams Isosweet (Staley 71% solids solution containing 42% fructose, 50% glucose, 1.5% maltose and 1.5% isomaltose) was combined in the glass jacketed reaction vessel with 1 gram DL-aspartic acid and 37.6 grams concentrated ammonium hydroxide. Steam was passed through the jacket until the internal temperature reached 94°C. The time required to reach the desired temperature was 15 minutes. The final pH of the reaction mixture was 5.3.

The reaction mixture was cooled to room temperature and an aliquot of undiluted reaction flavor product was sprayed on two batches of flue-cured tobacco stems to a level of 4% by weight and 2.6% by weight respectively. Cigarettes were made using 100% of the treated stem material. Controls were made using identical fluecured stems having no reaction flavor added thereto. Cellulose acetate filters were attached to all cigarettes prior to smoking. The cigarettes were smoked by an experienced panel of smokers. The cigarettes having 4% reaction flavor added thereto were found to have a burnt character or off-taste, were slightly peppery, and had a hot after-taste. The smokers also noted a slight paper character. The cigarettes treated at the 2.6% weight level had less stemmy character and hotness and were less papery than the cigarettes treated at the 4% level.

Example III

The following ingredients were premixed and added to a reaction vessel containing 50 ml concentrated ammonium hydroxide as in Example I.

426 grams Isomerase 900

1.3 grams glutamine

The contents of the vessel were stirred and heated to 95°C and then cooled. An aliquot of the cooled solution was diluted 1:1 with water and 10 µl portions were injected into a conventionally blended cigarette containing 20% by weight of the blend of unflavored, uncased reconstituted tobacco. The treated cigarettes were smoked and compared to untreated controls containing the same blend. The treated cigarettes were found to be generally smoother, less hot and peppery, less burnt and slightly sweet. An off-note was noted.

Flue-cured stems were sprayed to a weight level of 2.5% with an undiluted aliquot of the reaction flavor. Propylene glycol and glycerine were each sprayed on the same stems to a 2% weight level. On smoking, cigarettes containing the treated stems were found to have a smoother, slightly burnt and bitter taste as compared to the controls.

Example IV

In a manner identical to Example III, 1.3 grams glutamic acid was substituted for glutamine and the mixture was heated to 95°C and cooled. An

aliquot of the reaction product was diluted 1:1 and 10 µl portions were injected into cigarettes identical to those of Example III. On smoking, the treated cigarettes were found to be smoother, less bitter, more peppery and salty. Flue-cured stems were treated as in Example III. On smoking, the treated stems were smoother, slightly sweet, had less burnt taste and were not as harsh. A slightly bitter to sweet aftertaste was noted by some of the smokers.

Example V

Two reaction flavors (A and B) were prepared in which the reactants were combined, heated to 93°C in a reaction vessel and then cooled to room temperature. The reactants were as follows:

A. 361 grams 900 Isomerase; 1.3 g L-glutamic acid; 50 ml concentrated NH_4OH ;

B. 542 grams 900 Isomerase; 1.3 g L-glutamic acid; 50 ml concentrated NH_4OH ;

Undiluted reaction flavors A and B were sprayed on flue-cured bright stems to a level of 2.5% by weight of the stems. Glycerine and polyethylene glycol were sprayed on the flavored stems at a 2% weight level. On smoking 100% stem cigarettes, a smoother, slightly green papery or burnt taste was noted for reaction flavor A. Cigarettes containing reaction flavor B were slightly harsh, papery and had a green, burnt taste.

Claims

1. A process of producing a cooked flavorant in which a reducing sugar is heated with ammonia and an amino characterised in that the reducing sugar is reacted with a source of ammonia in a weight ratio of reducing sugar to ammonia calculated as ammonium hydroxide (30%) from 5 to 15:1, in the presence of only a trace amount of an amino acid or monoamide thereof in a weight ratio of sugar to amino acid from 200 to 300:1, and in the absence of any further reactant.

2. A process according to claim 1, characterised in that the weight ratio of reducing sugar to ammonium hydroxide (30%) is 6 to 8:1 (or a corresponding ratio for an ammonium salt), and that the weight ratio of the sugar to amino acid is from 235 to 245:1.

3. A process according to claim 1 or 2, characterised in that the mixture is heated to a temperature of 90 to 105°C and then either maintained at that temperature for 5 to 15 minutes or allowed to cool to room temperature.

4. A process according to claim 1, 2 or 3, characterised in that the amino acid or monoamide is selected from aspartic acid, glutamic acid, asparagine and glutamine.

5. A process according to any of claims 1 to 4, characterised in that the reducing sugar is selected from fructose, glucose, mannose, galactose and mixtures thereof.

6. A process according to any of claims 1 to 5, characterised in that the reaction product is cooled by diluting with water to effect a dilution of 1 to 5-1.

7. A smoking composition comprising a smokable filler material and a cooked flavorant reaction product produced by the process of any preceding claim.

8. A smoking composition according to claim 7 characterised by a filler of blended tobaccos containing from 0.5 to 10% by weight of cooked flavorant.

9. A smoking composition according to claim 7, characterised by a filler of tobacco and/or reconstituted tobacco and tobacco stems, wherein tobacco stems comprise up to 50% by weight of the filler and contain up to 5% of the cooked flavorant by weight of the stems.

10. A smoking composition according to claim 7, characterised by a filler of non-tobacco smoking substitute containing between 0.5 to 10% by weight of the cooked flavorant.

Patentansprüche

1. Verfahren zur Herstellung eines gekochten Aromastoffes, bei dem ein reduzierender Zucker mit Ammoniak und einer Aminosäure erhitzt wird, dadurch gekennzeichnet, dass der reduzierende Zucker mit einer Ammoniakquelle in einem Gewichtsverhältnis des reduzierenden Zuckers zu Ammoniak berechnet als Ammoniumhydroxid (30%), von 5 bis 15:1 in Anwesenheit von nur einer Spurenmenge einer Aminosäure oder eines Monoamids davon in einem Gewichtsverhältnis von Zucker zu Aminosäure von 200 bis 300:1 und in Abwesenheit eines weiteren Reaktionspartners umgesetzt wird.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass das Gewichtsverhältnis des reduzierenden Zuckers zu Ammoniumhydroxid (30%) 6 bis 8:1 beträgt (oder ein entsprechendes Verhältnis für ein Ammoniumsalz) und dass das Gewichtsverhältnis des Zuckers zur Aminosäure 235 bis 245:1 ist.

3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, dass die Mischung auf eine Temperatur von 90 bis 105°C erhitzt wird und dann entweder 5 bis 15 Minuten bei dieser Temperatur gehalten wird oder sich auf Raumtemperatur abkühlen kann.

4. Verfahren nach Anspruch 1, 2 oder 3, dadurch gekennzeichnet, dass die Aminosäure oder das Monoamid ausgewählt wird aus Asparaginsäure, Glutaminsäure, Asparagin und Glutamin.

5. Verfahren nach einem der Ansprüche 1 bis 4, dadurch gekennzeichnet, dass der reduzierende Zucker ausgewählt wird aus Fructose, Glucose, Mannose, Galactose und Gemischen davon.

6. Verfahren nach einem der Ansprüche 1 bis 5, dadurch gekennzeichnet, dass das Reaktionsprodukt durch Verdünnen mit Wasser, zur Bewirkung einer Verdünnung von 1 bis 5:1, gekühlt wird.

7. Rauchbare Zusammensetzung, enthaltend ein rauchbares Füllstoffmaterial und ein gekochtes Aromastoff-Reaktionsprodukt, hergestellt nach dem Verfahren eines der vorhergehenden Ansprüche.

8. Rauchbare Zusammensetzung nach Anspruch 7, gekennzeichnet durch einen Füllstoff aus gemischten Tabaken, enthaltend 0,5 bis 10 Gew.-% gekochten Aromastoff.

9. Rauchbare Zusammensetzung nach Anspruch 7, gekennzeichnet durch einen Füllstoff aus Tabak und/oder rekonstituiertem Tabak und Tabakstengeln, worin die Tabakstengel bis zu 50 Gew.-% des Füllstoffes betragen, und bis zu 5% des gekochten Aromastoffes, bezogen auf das Gewicht der Stengel, enthalten.

10. Rauchbare Zusammensetzung nach Anspruch 7, gekennzeichnet durch einen Füllstoff aus Nicht-Tabak-Raucherersatz, enthaltend zwischen 0,5 und 10 Gew.-% des gekochten Aromastoffes.

Revendications

1. Procédé de production d'un aromatisant cuit, selon lequel on chauffe un sucre réducteur avec de l'ammoniac et un amino-acide, caractérisé en ce qu'on fait réagir le sucre réducteur avec une source d'ammoniac suivant un rapport en poids du sucre réducteur à l'ammoniac, calculé en tant qu'hydroxyde d'ammonium (à 30%), qui est de 5 à 15:1, en présence de seulement une trace d'un amino-acide ou d'un monoamide de ce dernier, suivant un rapport en poids du sucre à l'acide qui est de 200 à 300:1 et en l'absence de tout autre corps participant à la réaction.

2. Procédé selon la revendication 1, caractérisé en ce que le rapport en poids du sucre réducteur à l'hydroxyde d'ammonium (à 30%) est de 6 à 8:1 (ou un rapport correspondant pour un sel d'ammonium), et en ce que le rapport en poids du sucre à l'acide est de 235 à 245:1.

3. Procédé selon la revendication 1 ou 2. carac-

térisé en ce qu'on porte le mélange à une température de 90 à 105°C puis, soit on le maintient à cette température pendant 5 à 15 minutes, soit on le laisse se refroidir jusqu'à la température ambiante.

4. Procédé selon la revendication 1, 2 ou 3, caractérisé en ce qu'on choisit l'acide ou son monoamide parmi l'acide aspartique, l'acide glutamique, l'asparagine et la glutamine.

5. Procédé selon l'une quelconque des revendications 1 à 4, caractérisé en ce qu'on choisit le sucre réducteur parmi le fructose, le glucose, le mannose, le galactose et des mélanges de ceux-ci.

6. Procédé selon l'une quelconque des revendications 1 à 5, caractérisé en ce qu'on refroidit le produit de réaction en le diluant avec de l'eau de manière à réaliser une dilution de 1 à 5:1.

7. Composition à fumer comprenant un matériau de remplissage fumable et un produit de réaction aromatisant cuit produit par un procédé selon l'une quelconque des revendications précédentes.

8. Composition à fumer selon la revendication 7, caractérisée par un remplissage de tabacs mélangés contenant de 0,5 à 10% en poids d'aromatisant cuit.

9. Composition à fumer selon la revendication 7, caractérisée par un remplissage de tabac et/ou de tabac reconstitué et de tiges de tabac, dans lequel ces tiges de tabac représentent jusqu'à 50% en poids du remplissage et contiennent jusqu'à 5% de l'aromatisant cuit par rapport au poids des tiges.

10. Composition à fumer selon la revendication 7, caractérisée par un remplissage de substitut à fumer sans tabac contenant entre 0,5 et 10% en poids d'aromatisant cuit.

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